National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1620c

Sulfur in Residual Fuel Oil (4 %)

This Standard Reference Material (SRM) is intended for use in the calibration of instruments and the evaluation of methods used in the determination of total sulfur in fuel oils or materials of similar matrix. A unit of SRM 1620c consists of 100 mL of commercial "No. 6" residual fuel oil as defined by ASTM D 396-97 Standard Specification for Fuel Oils [1].

Certified Value: The certified sulfur content reported in Table 1 is based on determinations by isotope dilution thermal ionization mass spectrometry (ID-TIMS) [2]. Homogeneity testing was performed using X-ray fluorescence (XRF) spectrometry. The uncertainty in the certified value is expressed as an expanded uncertainty and is calculated according to the method in the ISO and NIST Guides [3]. The expanded uncertainty is computed at the 95 % level of confidence.

Table 1. Certified Value (mass fraction)

Sulfur: 4.561 % ± 0.015 %

Information Values: The information values reported in Table 2 are noncertified values with no uncertainty assessed. They are provided as supplemental information to better characterize the matrix.

ASTM Information: SRM 1620c was included as an unknown in the May 1998, ASTM Committee D-2 Interlaboratory Crosscheck Program for No. 6 Fuel Oil as Sample ID: #6F9805. Summary statistics reported by ASTM are provided in the addendum to this certificate to demonstrate user experience with this material using ASTM methods and to better characterize the matrix. The ASTM Committee D-2 Interlaboratory Crosscheck results were not used in calculating the certified sulfur value for SRM 1620c and should not be used as a substitute for the NIST certified value.

Expiration of Certification: The certification of this SRM is valid until **01 October 2010**, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Use and Handling"). However, the certification will be nullified if the SRM is damaged, contaminated, or modified.

The overall direction and coordination of the technical measurements leading to certification of this SRM were directed by J.D. Fassett of the NIST Analytical Chemistry Division. Analytical measurements were performed by W.R. Kelly, R.D. Vocke, Jr., A.F. Marlow, J.R. Sieber, and J.L. Mann of the NIST Analytical Chemistry Division.

Statistical consultation for this SRM was provided by K.R. Eberhardt of the NIST Statistical Engineering Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 01 March 2006
See Certificate Revision History on Last Page

Robert L. Watters, Jr., Chief
Measurement Services Division

NOTIFICATION TO USERS

Stability: This material is considered to be stable during the period of certification. NIST will monitor this material and will report any significant changes in certification to the purchaser. Registration (see attached sheet) will facilitate notification.

Use and Handling: Because of the viscosity of SRM 1620c, it is recommended that the SRM unit be warmed slowly to between 40 °C and 60 °C and then shaken, or stirred with a clean stirrer, before sampling. Care must be exercised to **NOT** introduce entrapped air that could affect gravimetric measurements and XRF responses. A detailed study to determine if the sulfur components of SRM 1620c will segregate has not been performed at this time.

The SRM bottle should only be opened for the minimum time required to dispense the material. To relate analytical determinations to the certified value in this Certificate of Analysis, a minimum sample mass of 140 mg should be used. After use, the bottle should be tightly recapped and stored under normal laboratory conditions away from direct sunlight.

SUPPLEMENTAL INFORMATION

Information Values: The information values provided in Table 2 are based on results by a commercial firm under contract to NIST using ASTM methods. They are given as additional information on the matrix only.

Table 2. Information Values

Measurement		ASTM Standard Used	Result
Kinematic Viscosity	@ 40 °C	D 445-94	7131 × 10^{-6} m^2/s (7131 cSt)
	@ 50 °C	D 445-94	2982 × 10^{-6} m^2/s (2982 cSt)
	@ 100 °C	D 445-94	150.9 × 10^{-6} m^2/s (150.9 cSt)
Carbon (mass fraction)		D 5291-92	84.9 %
Hydrogen (mass fraction)		D 5291-92	10.7 %

REFERENCES

[1] ASTM D 396-95, *Standard Specification for Fuel Oils*, *Annu. Book ASTM Stand.*, Vol. 05.01, West Conshohocken, PA (1997).

[2] Kelly, W.R.; Paulsen, P.J.; Murphy, K.E.; Vocke, R.D., Jr.; Chen, L.-T.; *Determination of Sulfur in Fossil Fuels by Isotope Dilution Thermal Ionization Mass Spectrometry*, Anal. Chem., Vol. 66, pp. 2505-2513 (1994).

[3] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, lst ed. International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at http://physics.nist.gov/Pubs/.

Certificate Revision Date History: 01 March 2006 (Editorial changes); 22 March 2000 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Telephone (301) 975-6776; Fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

Addendum

Standard Reference Material® 1620c

Sulfur in Residual Fuel Oil

ASTM Committee D-2 Interlaboratory Crosscheck Program Results: SRM 1620c was included as an unknown in the May 1998, ASTM Committee D-2 Interlaboratory Crosscheck Program for No. 6 Fuel Oil as Sample ID: #6F9805 [1]. Summary statistics as reported by ASTM are provided in the addendum to this certificate to demonstrate user experience with this material using ASTM methods and to better characterize the matrix. The ASTM Committee D-2 Interlaboratory Crosscheck results were not used in calculating the certified sulfur value for SRM 1620c and should not be used as a substitute for the NIST certified value.

Gaithersburg, MD 20899
Issue Date: 22 March 2000

Summary Statistics Reported in the May 1998 ASTM Committee D-2 Interlaboratory Crosscheck Program for No. 6 Fuel Oil

Analyte	No. Valid Results	Robust[a] Mean	Robust[a] Standard Deviation	Reproducibility ASTM Standard	Reproducibility[a] These Test Data	Comments
Aluminum D 5184 (mg/kg) Method A: ICP	22	9.6	4.6	3.2	12.7	
Aluminum D 5184 (mg/kg) Method B: AAS	38	9.5	5.3	3.5	14.7	
API Gravity D 287 (°API)	84	7.88	0.38	0.50	1.05	
Ash D 482 (mass %)	106	0.0590	0.0107	0.0050	0.0296	The data from two laboratories were not included in statistical analysis.
Asphaltenes D 3279 (NHI, %)	63	16.241	1.921	2.780	5.321	
Carbon Residue (Conradson) D189 (mass %)	49	22.726	1.041	3.474	2.884	
Carbon Residue (Micro Method) D 4530 (mass %)	73	23.742	0.604	2.027	1.673	
Density D 1298 (kg/L) at 15 °C	91	1.01505	0.00258	0.00120	0.00715	Labs were instructed to report results to 15 °C. Labs results initially developed in 60 °F were instructed to convert their data to 15 °C using Petroleum Measurement Tables (Guide 1250). The data from one lab was converted to kg/L.
Flash Point of Ordinary Liquids D 93 (°C) Manual/Automatic Procedure A (Corrected Flash Point)	119	80.24	5.43	6.26	15.04	The data from five laboratories were converted to °C. The results from 31 laboratories were initially developed in °F.
Heat Content D 240 (MJ/kg)	47	41.4600	0.3658	0.4000	1.0133	The data from one laboratory were not included in statistical analysis.
Nitrogen D 3228 (mass %)	22	0.4091	0.0847	0.1215	0.2346	
Nitrogen D 4629 (mg/kg)	19	3041.482	1660.860	See Comments	4600.582	ASTM D 4629 is applicable to naphthas, distillates, and oils containing (0.3 to 100) mg/kg total nitrogen. The Robust Mean is above this range, therefore ASTM Reproducibility not determined. The data from two laboratories were not included in statistical analysis.

Analyte	No. Valid Results	Robust[a] Mean	Robust[a] Standard Deviation	Reproducibility ASTM Standard	Reproducibility[a] These Test Data	Comments
Nitrogen D 5291 (mass %)	7	0.4366	0.1870	See Comments	0.5180	The Reproducibility in D 5291 addresses nitrogen in the concentration range of (0.75 to 2.5) mass %. The Robust Mean of these data is below this range, therefore ASTM Reproducibility not determined.
Nitrogen D 5762 (μg/g)	10	4502.050	694.871	1310.097	1924.793	
Pour Point D 97 (°C) Manual/Automatic	97	13.9	6.2	6.0	17.2	ASTM D 97 states to report pour point limits at temperatures in multiples of 3 °C. Test data from labs not reporting in multiples of 3 °C or results initially developed in °F are not included in the statistical analysis or graphs. Ten labs reported that their test results were initially developed in °F. Five labs reported test results that were not multiples of three.
Sediment D 473 (mass %)	83	0.024	0.014	0.039	0.039	
Sediment D 4870 (m/m)	27	0.031	0.022	0.060	0.061	
Silicon D 5184 (mg/kg) Method A: ICP	22	11.0	5.6	3.7	15.5	
Silicon D 5184 (mg/kg) Method B: AAS	35	14.6	10.1	8.0	28.0	
Sulfur D 129 (mass %)	4	4.4250	0.2786	0.2700	0.7717	
Sulfur D 1552 (mass %) Iodate Procedure	4	4.1880	1.4989	0.5400	4.1520	
Sulfur D 1552 (mass %) IR Procedure	5	4.6410	0.0836	0.4900	0.2316	
Sulfur D 2622 (mass %)	18	4.47469	0.14825	0.71595	0.41065	
Sulfur D 4294 (mass %)	96	4.4789	0.1666	0.3200	0.4615	
Vanadium D 1548 (mg/kg)	14	215.497	32.332	21.550	89.560	
Vanadium D 5708 (mg/kg) Method A: ICP with an Organic Solvent Specimen Solution	16	203.2717	22.9060	33.5725	63.4496	
Nickel D 5708 (mg/kg) Method A: ICP with an Organic Solvent Specimen Solution	17	57.3923	6.3715	11.7457	17.6491	

Analyte	No. Valid Results	Robust[a] Mean	Robust[a] Standard Deviation	Reproducibility ASTM Standard	Reproducibility[a] These Test Data	Comments
Iron D 5708 (mg/kg) Method A: ICP with an Organic Solvent Specimen Solution	14	15.1421	5.7439	See Comments	15.9106	The Reproducibility in D 5708 addresses iron in the concentration range of (1 to 10) mg/kg. The Robust Mean is above this range, therefore ASTM Reproducibility not determined.
Vanadium D 5708 (mg/kg) Method B: ICP after Acid Decomposition	3	210.2500	24.8884	28.5108	68.9409	
Nickel D 5708 (mg/kg) Method B: ICP after Acid Decomposition	9	62.0714	7.1725	4.3593	19.8678	
Iron D 5708 (mg/kg) Method B: ICP after Acid Decomposition	9	24.8571	6.0012	See Comments	16.6233	The Reproducibility in D 5708 addresses iron in the concentration range of (1 to 10) mg/kg. The Robust Mean is above this range, therefore ASTM Reproducibility not determined.
Vanadium D 5863 (mg/kg) Method A: AAS (Decomposed with Acid)	15	217.3045	33.8505	46.7761	93.7659	
Nickel D 5863 (mg/kg) Method A: AAS (Decomposed with Acid)	24	212.7250	32.6897	45.8880	90.5505	
Nickel D 5863 (mg/kg) Method A: AAS (Decomposed with Acid)	23	60.4823	7.3742	10.1077	20.4265	
Iron D 5863 (mg/kg) Method A: AAS (Decomposed with Acid)	27	29.2840	10.5952	See Comments	29.3487	The Reproducibility in D 5863 addresses iron in the concentration range of (3 to 10) mg/kg. The Robust Mean is above this range, therefore ASTM Reproducibility not determined.
Vanadium D 5863 (mg/kg) Method B: AAS (Sample Diluted with an Organic Solvent)	25	235.8899	34.7480	160.9539	96.2520	
Nickel D 5863 (mg/kg) Method B: AAS (Sample Diluted with an Organic Solvent)	21	56.0415	20.0146	4.2854	55.4404	The data from one laboratory were not included in statistical analysis.
Sodium D 5863 (mg/kg) Method B: AAS (Sample Diluted with an Organic Solvent)	32	17.2389	4.0595	11.8948	11.2448	

Analyte	No. Valid Results	Robust[a] Mean	Robust[a] Standard Deviation	Reproducibility ASTM Standard	Reproducibility[a] These Test Data	Comments
Viscosity, Kinematic D 445 (mm^2/s) 50 °C	70	6524.1458	554.9784	482.7868	1537.2902	Twenty-three labs reported that their test results were initially developed in °F.
Viscosity, Kinematic D 445 (cSt) 100 °C	87	240.7132	15.2107	9.9485	42.1336	Twelve labs reported that their test results were initially developed in °F.
Water and Sediment D 1796 (vol %)	87	0.067	0.042	0.110	0.116	
Water Content D 95 (mL)	82	0.06	0.04	0.20	0.11	

[a] The algorithms used to calculate the robust statistical summaries used in this table are described in the ASTM report [1] and are based on [2].

ASTM Standards

D 93-94	Standard Test Methods for Flash Point by Pensky-Martens Closed Tester
D 95-83(1990)	Test Method for Water in Petroleum Products and Bituminous Materials by Distillation
D 97-96a	Test Method for Pour Point of Petroleum Products
D129-95	Test Method for Sulfur in Petroleum Products (General Bomb Method)
D189-97	Test Method for Conradson Carbon Residue of Petroleum Products
D 240-92 (1997) [,1]	Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter
D 287-92 (1995)	Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)
D 445-96	Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)
D 473-81 (1995) [,1]	Practice for Sediment in Crude Oils and Fuel Oils by the Extraction Method
D 482-95	Test Method for Ash From Petroleum Products
D 1298-85 (1990) [,1]	Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method
D 1548-92 [,1]	Test Method for Vanadium in Navy Special Fuel Oil
D 1552-95	Test Method for Sulfur in Petroleum Products (High-Temperature Method)
D 1796-97	Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)
D 2622-94	Test Method for Sulfur in Petroleum by X-Ray Spectrometry
D 3228-96	Test Method for Total Nitrogen in Lubricating Oils and Fuel Oils by Modified Kjeldahl Method
D 3279-90	Test Method for n-Heptane Insolubles
D 4294-90 (1995) [,1]	Test Method for Sulfur in Petroleum Products by Energy-Dispersive X-Ray Fluorescence Spectroscopy
D 4530-93	Test Method for Determination of Carbon Residue (Micro Method)
D 4629-96	Test Method for Trace Nitrogen in Liquid Petroleum Hydrocarbons by Syringe/Inlet Oxidative Combustion and Chemiluminescence Detection
D 4740-95	Test Method for Stability and Compatibility of Residual Fuels by Spot Test
D 4870-94	Test Method for Determination of Total Sediment in Residual Fuels
D5184-91 (1995)	Test Method for Determination of Aluminum and Silicon in Fuel Oils by Ashing, Fusion, Inductively Coupled Plasma Atomic Emission Spectrometry and Atomic Asorption Spectrometry
D 5291-96	Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Petroleum Products and Lubricants
D 5453-93	Test Method for Determination of Total Sulfur in Light Hydrocarbons, Motor Fuels and Oils by Ultraviolet Fluorescence
D 5708-95a	Test Methods for Determination of Nickel, Vanadium, and Iron in Crude Oils and Residual Fuels by Inductively Coupled Plasma (ICP) Atomic Emission Spectrometry
D 5762-95	Test Method for Nitrogen in Petroleum and Petroleum Products by Boat/Inlet Chemiluminescence
D 5863-95	Test Method for Determination of Nickel, Vanadium, Iron, and Sodium in Crude Oils and Residual Fuels by Flame Atomic Absorption

[,1] Indicates that only editorial changes were made to the previous issuance of the ASTM standard.

REFERENCES

[1] "ASTM Committee D-2 Interlaboratory Crosscheck Program for No. 6 0.1 Sample ID: #6F9805, May 1998," ASTM, West Conshohocken, PA, (1998).

[2] "Robust Statistics – How Not to Reject Outliers," by the Analytical Methods Committee of the Royal Society of Chemistry, *Analyst*, 114, pp. 1693-1697, (Dec. 1989).